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Qualitative and Quantitative Analysis of Mixtures of Compounds Containing Both Hydrogen and Deuterium

Investigations of compounds with partially deuterated ethyl groups have resulted in a general method for the qualitative and quantitative analysis of mixtures of partially deuterated compounds. Deuterium isotope effects are already in wide use today for structural and kinetic studies in organic chemistry and biochemistry. It is suggested that new areas of study in biosynthesis and intermediary metabolism may be opened by this general method of analysis. Of particular interest is the possibility of investigating biosynthetic pathways, using fully deuterated organisms introduced to an ordinary hydrogen substrate.

In general, the method incorporates two well known analytical techniques. Nuclear magnetic resonance (NMR) spectroscopy has been widely used for the determination of the location and amount of deuterium in organic compounds. The technique is accurate for partially deuterated compounds, but fully deuterated compounds show no magnetic resonance; hence, determination of relative amounts of fully deuterated components in a mixture, by this technique, is indirect and therefore not very accurate.

Mass spectroscopy is the other method presently used in the analysis of isotopically substituted compounds. The mass spectrum of a mixture of partially deuterated compounds will provide the relative amounts of components according to their molecular formulas. With this technique, it is possible to detect directly the fully deuterated species, and, with a suitable instrument, it is possible to carry out measurements with an accuracy that exceeds NMR measurements. However, no reliable information about the

location of deuterium can be obtained from mass spectra.

Neither NMR spectroscopy or mass spectroscopy alone can be used for a complete and accurate analysis of a mixture of compounds containing both hydrogen and deuterium. However, the results of these two techniques can be combined to provide highly accurate and reliable values for the composition of the mixture. This procedure was used for quantitative analysis of ethyl acetate isotopic mixtures. It was found that errors could easily be detected and the reliability of the data can be internally checked. In a mixture of partially deuterated ethyl acetates, all of the isotopic components can be determined with a relative error of $\pm 1.5\%$.

Notes:

1. NMR spectra were recorded on a Varian HA 100 spectrometer at 100 MHz. Mass spectral data were collected on a mass spectrometer with an analyzer tube of 12-inch radius and 60° sector, an Atlas To-4 ion source, and an electron multiplier ion collector.
2. Experimental methods and materials and a discussion of results are contained in *Analytical Biochemistry*, vol. 22, 1968, "Qualitative and Quantitative Isotopic Analysis of Ethyl Groups Containing Both Hydrogen and Deuterium ($\text{CH}_x\text{D}_{3-x}\text{CH}_y\text{D}_{2-y}$) by Combined NMR and Mass Spectroscopy," W. Saur, H.L. Crespi, L. Harkness, G. Norman, and J.J. Katz.
3. This information may be of interest to drug companies, fermentation companies, and such organizations as the National Institutes of Health and the

(continued overleaf)

U.S. Food and Drug Administration.

4. Inquiries may be directed to:

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Patent status:

Inquiries concerning rights for commercial use of
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